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OIL SORPTION CAPABILITY OF TREATED HUMAN HAIR SORBENT FOR OIL SEA-WATER CLEANUP

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Highlights:

- Explore on the human hair for oil spill clean-up.
- Assessing the effect of hot water treatment and mercerization process on human hair.
- Evaluate the effect of different alkaline and its concentration towards sorption capacity.
- Comparing the modified and unmodified human hair for oil sorption capacity.

Abstract: This study explored the use of modified human hair as a biosorbent for oil spill clean-up, with a focus on enhancing its oil sorption capacity. Human hair was modified using Sodium Hydroxide (NaOH) and Potassium Hydroxide (KOH) through two methods: hot water treatment at 80°C and mercerization with 5% NaOH and KOH. Structural analysis was conducted, including elemental composition, surface roughness (via Field Emission Scanning Electron Microscopy or FESEM), and hydrophobicity (via wettability tests). The oil sorption capacity was tested using an oil-seawater mixture at different adsorption times (60-100 minutes). Results showed that hair treated with hot water at 100°C achieved the highest oil sorption capacity (2.592 g/g), followed by hair treated with 5% NaOH (2.471 g/g). The optimal adsorption time was found to be 80 minutes, with all samples showing increased oil sorption. The study concluded that hot water treatment significantly improved the surface roughness and hydrophobic properties of the hair, leading to enhanced oil sorption. In comparison, mercerization with NaOH and KOH was less effective in improving the sorption capacity. Raw human hair showed high oil sorption initially, but its effectiveness decreased over extended adsorption times. These findings suggest that modified human hair, especially when treated with hot water, has strong potential for use in oil spill clean-up due to its improved structural and hydrophobic properties.

Keywords: human hair; mercerization; oil sorption; heat treatment; biosorbent

1. Introduction

Oil spills severely degrade environmental quality, endanger health, contaminate water, and devastate natural resources (Ifelebuegu *et al.*, 2015). According to Che Ishak & Arof (2019), who have summarised the incidents of oil spills, the causes of the spills may be divided into four categories: human, technical, natural, and occasionally unidentified sources. A recent incident in Malaysia on February 11, 2023, caused a four-hour outage at several water treatment plants, affecting water supply for 1,337 areas in Selangor and Kuala Lumpur (Chen, 2023). Clean-up involved oil booms and pads, but larger spills require more effective methods. The Deepwater Horizon spill in the Gulf of Mexico on April 20, 2010, lasted three months, resulting in 11 deaths and 17 injuries, and was cleaned using various techniques including natural weathering, dispersants, and biosorbents (Al-Majed, Adebayo, & Hossain, 2012).

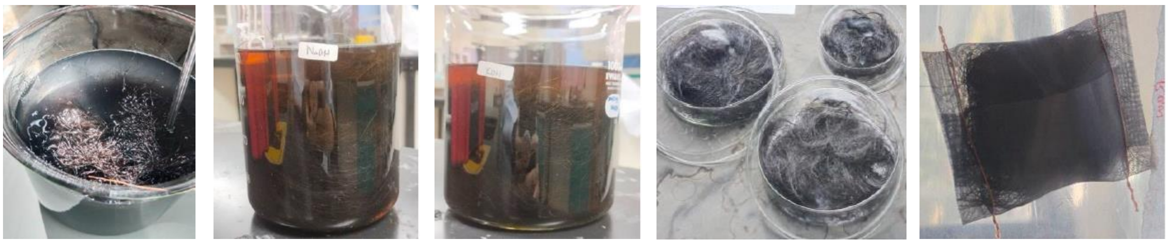
In recent years, the world who are still reliant on fossil fuel products had been researching more clean-up methods especially those that are eco-friendly and cost-effective due to the limitations of the traditional methods such as chemical dispersant, boom, and bioremediation in terms of cost, eco-friendliness, labour, intensity, and area of application (Tan *et al.*, 2021). Due to the limitations of traditional methods, research has focused on eco-friendly, cost-effective clean-up techniques. The Deepwater Horizon spill notably used sorbents, including peat moss, which is eco-friendly and cost-effective (Ogunbiyi *et al.*, 2023). Other biosorbents used include kapok, rice husk, coconut husk, barley straw, cotton grass, sugarcane bagasse, sawdust, wool fiber, walnut shell, feathers, garlic peel, activated carbon, and human hair. The use of human hair as a biosorbent was pioneered by hairstylist Philip McCrory in 1993. According to Sharma (2016), raw human hair had an oil spill recovery of 88% and when the used human hair was disposed, the oil can subsequently be broken down by oyster mushrooms that can be grown on the greasy hair making it a good compost material.

According to Gupta (2014), on average, human hair outperformed other sorbent materials such as regenerated cellulose, polypropylene, and cotton by-products in terms of crude oil adsorption since compared to other products, booms manufactured with hair sorbent exhibited a greater range in oil absorption, which is probably related to the non-uniform nature of mixed human hair. Human hair can be reused after wringing out the oil, making it advantageous over other methods. Researchers suggest modifying human hair could further enhance its pollutant absorption capabilities (Oliveira *et al.*, 2020). This study investigates the effectiveness of modified human hair in oil adsorption through hot water treatment, mercerization process with NaOH and KOH at different adsorption time. This research aims to compare the effective of mercerization process in hair modification of using NaOH and KOH, which is thus varies in concentration for either NaOH or KOH based on the oil sorption result obtained, in different adsorption time.

2. Materials and Methods

2.1. Hot Water Treatment and Mercerization Modification Process

A 200 g of raw human hair was collected from a salon which was then washed with detergent and tap water to remove wax, then rinsed and dried under natural sunlight for 48 hours before being stored in a plastic bag. Next, 30 g of this hair was immersed in 800 mL of 80°C distilled water for 1 hour as for the hot treated human hair sample (HTH). The process was conducted at room temperature (25°C and 1 atm). Mercerization involved soaking 30 g of hair in two different alkaline solutions: Sodium Hydroxide, NaOH and Potassium Hydroxide, KOH. Each solution was prepared by diluting 40 mL of a 1 mol alkaline solution with 760 mL of distilled water to make 800 mL of a 5% solution. The hair was soaked for 48 hours, then dried under sunlight for another 48 hours. The raw and treated hair samples were then placed in mesh wire casings (0.05 mm pore size) with about 2g of hair each. This encasement minimized errors in oil sorption capacity measurement by preventing hair loss during the experiment. The overall modification conducted is shown in **Figure 1**.



(**a**) (**b**) (**c**) (**d**) (**e**)

Figure 1. Hair modification: (a) HTH sample; (b) NaOH mercerization; (c) KOH mercerization; (d) hair drying after modification; (e) hair modified sorbent with casing

2.2. Structural characterization

This research analysed the structural and oil sorption characteristics of the raw and modified hair samples. Structural analysis examined the correlation between hair surface roughness and sorption capacity, hypothesizing that increased roughness would enhance hydrophobicity. The sorption capacity was assessed through a wettability test to see if seawater adhered to modified hair samples. Field Emission Scanning Electron Microscopy with energy dispersive X-ray spectroscopy (FESEM/EDX) was used to analyse surface roughness, pore size, and the hair's hydrophobic and oleophilic characteristics. Hair samples were sent to Pusat Instrumentasi dan Perkhidmatan Sains (PIPS) UMS, coated with t-butyl alcohol for FESEM analysis at X1000 magnification, while EDX analysis was conducted at X2000 magnification. The wettability test, using ImageJ Software with a contact angle analyze by the pictures of dropping seawater from UMS Outdoor Development Center (UMS ODEC) beach onto hair samples.

2.3. Oil Removal by Human Hair Sorbent by Adsorption

To achieve the second objective of this study, the oil sorption capacity method was used to measure the human hair sorbent's oil sorption capacity (Chenxi *et al.*, 2022). The calculation, shown in equation (1), uses Q (g/g) for oil absorbency, G_1 (g) for the hair sample's weight before adsorption, G_2 (g) for the hair sample's weight after adsorption, and G_0 (g) for the empty petri dish's weight. Diesel oil, purchased locally, and seawater from UMS ODEC beach were used. The test was conducted in the Drilling Lab at UMS under room conditions.

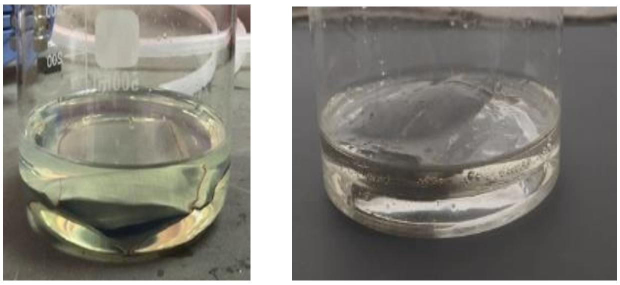
$$Q = \frac{G_2 - (G_0 + G_1)}{G_1} \tag{1}$$

2.4. Oil Removal by Human Hair Sorbent for Mass Loading

The mass loading performed on the raw human hair sample to determine contact time and control oil sorption capacity. The hair sample was immersed in 5 g of diesel oil for 10, 40, 60, 80, and 100 minutes (Varsha & Mohanan, 2023). After each contact time, the hair samples were removed for measurement. The best contact times were identified as before reaching maximum oil sorption, at maximum capacity, and when capacity declined. The optimum times were 60, 80, and 100 minutes. Mass loading on modified human hair was then conducted using these optimum times. The sample of oil mass loading test is shown in **Figure 2(a)**.

2.5. Oil-Seawater Removal by Human Hair Sorbent

Figure 2(b) shows the oil-seawater sorption test, which was conducted with contact times of 60, 80, and 100 minutes. Diesel oil concentration in seawater was 20% (Al-Majed *et al.*, 2023). Diesel oil (10 g) was mixed with seawater (50g) before immersing hair samples for their respective contact times. Afterward, the hair was removed from its mesh casing and vacuum filtered to remove most of the adsorbed water, ensuring mainly oil adhered to the hair. Hair samples tested included raw human hair, hair treated with 5% NaOH, 5% KOH, and 80°C hot water treated hair (HTH). The best-modified hair would be further tested by varying parameters to maximize oil sorption, achieving the third objective. Based on structural, compositional, and wettability analysis, 5% NaOH mercerized hair was selected. Afterward, 20% and 30% NaOH were tested, considering environmental concerns. Additional tests with 70°C and 90°C hot water treatments were conducted to investigate their relationship to oil sorption capacity.



(**a**) (**b**)

Figure 2. Oil sorption experiment: (a) pure oil mass loading; (b) oil-seawater

3. Results and Discussion

3.1. Modification on Human Hair

For this study, the first objective was achieved by preparing raw human hair samples and modifying them through mercerization and hot water treatment. To confirm the success of these modifications, visual, structural, and wettability analyses were conducted. Visual analysis showed that the 80°C hot water treatment resulted in only slight white murkiness, indicating some reaction had occurred. This murkiness suggests the presence of residual dirt, detergent, and slight hair breakage due to heat damage starting at 40°C (Zamani *et al.*, 2021). In contrast, mercerization showed clearer signs of modification. The alkaline solution's color changed from clear to brownish orange within 48 hours of immersion. This color change in dicates the removal of sulphur, lignin, and dirt, along with the breakage of cystine and OH bonds in the hair fibers. According to the breaking of cystine and sulphur gives a reddish-brown color. Additionally, a phenol odor, aligned with the effects of mercerization on biomaterial, was noted (Kathiresan & Meenakshisundaram, 2022).

3.2. Structural and Composition Characterization Analysis

The modification of human hair through heat treatment and mercerization will increase the surface roughness and wettability of the hair which simultaneously increase the adsorption capacity and hydrophobicity respectively (Wang, Zheng & Wang, 2012). The human hair modifications were proved analytically to achieve the first objective with structural and compositional analysis through FESEM+EDX which were done to determine the structural and compositional characteristics of oil sorption capabilities of the human hair samples. The human hair samples also undergone wettability analysis toward seawater to determine the change in hydrophobicity after modification of the human hair samples.

3.2.1. FESEM Analysis

Field Emission Scanning Electron Microscopy (FESEM) is one of the instruments used to analyse the surface morphology changes between raw and modified human hair were analysed. Raw human hair is smooth due to amino acids and cuticles, as seen in **Figure 3(a)**. Hair treated with NaOH showed a porous, rough surface with flakes as shown in **Figure 3(b)**, indicating successful modification. Similarly, KOH-treated hair exhibited a rough, porous surface as shown in **Figure 3(c)**. Zamani et al. (2021) stated that, the hot water at 40°C damages hair, forming cracks seen in SEM images. The FESEM image of 80°C hot water-treated hair in **Figure 3(d)** confirmed these findings, indicating successful modification. The rougher surfaces of the sorbent could improve liquid sorption due to the more pores and pits (Saleem *et al.*, 2023).

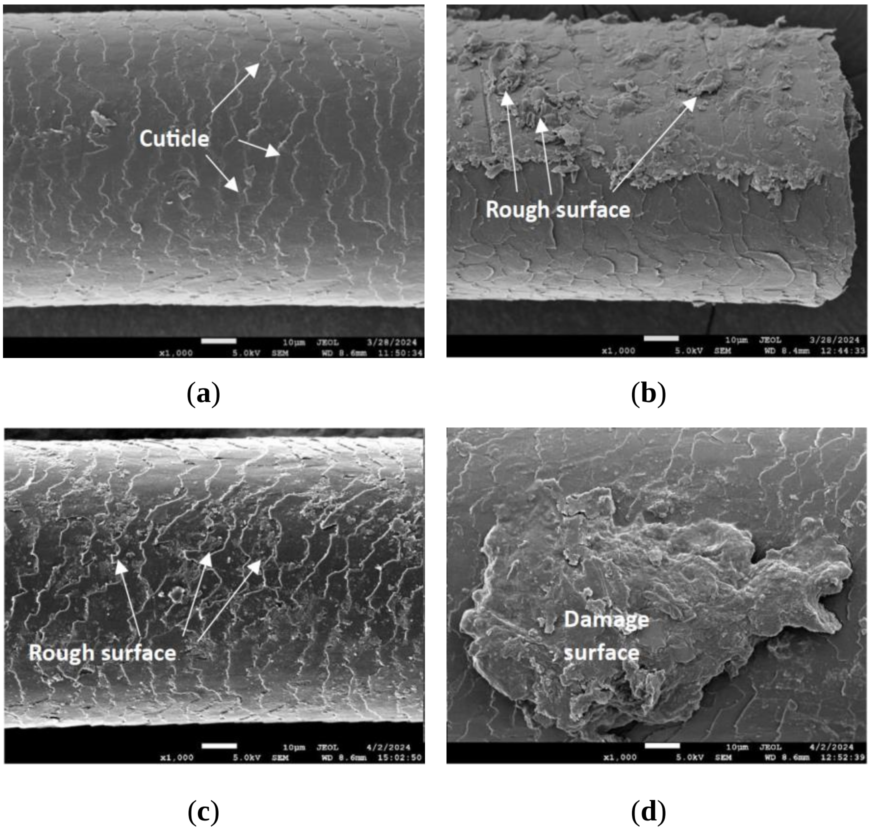


Figure 3. FESEM micrography with magnification of x1000 (5.0kV, 10 μm, WD 8.6 mm): (a) raw human hair; (b) 5% NaOH mercerized human hair; (c) 5% KOH mercerized human hair; (d) 80 °C HTH

In comparison between 5% NaOH and 5% KOH mercerized hair, NaOH treatment caused more severe surface damage with thicker flakes, while KOH treatment showed smaller pores with thin cuticle lifting. Both treatments increased surface roughness and sorption, but NaOH was more effective. Meanwhile, in comparison between 80°C hot water-treated hair and 5% NaOH-treated hair. Hot water treatment caused more severe but localized damage, creating large crevices, while NaOH treatment caused smaller, spread-out damage. Larger crevices in hot water-treated hair indicated better liquid adherence. It also showed that treated with hot water producing more rougher and coarser surface as compared to the mercerized sample. The coarser and undulant surface cannot only increase the fibre surface area but also improve the adhesion property of oil on the sorbent surface (Wang *et al.*, 2023).

3.2.2. EDX Analysis

The success of each human hair modification was analyzed using Energy Dispersive X-ray (EDX) quantification. Results from EDX are shown in **Table 1**. Based on the elemental composition of the referred study, which is Carbon (C), Oxygen (O), Sulphur (S), and Nitrogen (N) elements to this experiment raw human hair, the composition matched each other in types of elements (C, O, S, N) but have on average ±4.9 wt% difference in weight percentages value. However, the weight percentages of the elements were considered negligible since this study focused only on the presence of elements in the hair sample. Thus, the control sample which was the raw human hair of this experiment matched the theoretical composition of raw human hair with the presence of C, O, S, and N elements. This difference is negligible as the focus was on the presence of elements. The control sample matched the theoretical composition, containing C, O, S, and N elements. Mercerization introduced Natrium (Na) in 5% NaOH-treated hair and Potassium (K) in 5% KOH-treated hair, as shown in **Table 1**. This is due to element deposition during treatment. The 80°C hot water treatment did not alter the elemental composition, matching raw hair since it did not involve element deposition. The EDX analysis confirmed the success of mercerization with the respective alkaline.

Table 1. Elemental composition percentage of raw and modified human hair biosorbent

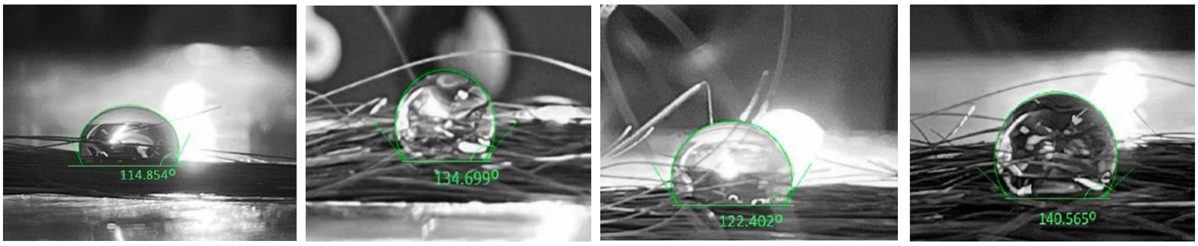
Type of sample	Elemental composition (%)						Reference
	C	O	N	S	Na	K	
Raw human hair	59.2	15.1	13.3	12.3	-	-	Coroaba *et al.*, 2020
Raw human hair	57.7	24.9	8.5	8.9	-	-	This experiment
5% NaOH mercerized-human hair	55.8	22.5	13.4	7	1.3	-	This experiment
5% KOH mercerized-human hair	62.0	19.7	14.2	2.8	-	1.3	This experiment
80 ºC HTH	62.5	19.6	15	2.9	-	-	This experiment

3.3. Wettability Analysis

The success of using modified human hair as an oil biosorbent was analyzed through wettability analysis using ImageJ Software. Seawater which was collected from the UMS ODEC beach was dropped on the hair samples using a syringe and the image of the droplet on the specimen were taken using a high resolution captured and then were upload and analysed using the ImageJ software manually. The result of the raw human hair contact angle was compared to the study by Chenxi *et al.* (2022). Results are shown in **Table 2** and **Figure 4**. The wetting behaviour of surfaces depends on their morphology (Chenxi et al., 2022). When water does not saturate a rough surface, air pockets form, increasing the contact angle and enhancing hydrophobicity (Saleh & Baig, 2019). The rough structures can make a smooth oleophilic surface more oleophilic (Liu et al., 2013). Raw human hair is naturally hydrophobic, with a contact angle of >90°C; pure water on dry untreated hair has a contact angle of ~98º, and wet untreated hair ~103º (Lodge & Bhushan, 2006). While there is no contact angle analysis for human hair with seawater, comparisons can still be made. The higher contact angle of human hair with seawater is due to foreign matter in seawater increasing surface tension (Sghaier & Ben Nasrallah, 2006). Modified human hair showed an increased contact angle due to greater surface roughness, with 80°C heat-treated hair showing the best oil sorption, followed by 5% NaOH and 5% KOH mercerized hair. This result complies to the research conducted by Vaišis *et al.* (2022), which increasing of sorbent hydrophobicity and oleophilic observed after application of its chemical modification and hot water treatment.

Table 2. Elemental composition percentage of raw and modified human hair bio-sorbent

Type of samples	Contact angle (°) of seawater on samples	Reference
Raw human hair	103.0 (pure water)	Wang et al, 2013
Raw human hair	114.9	This experiment
5% NaOH mercerized-human hair	134.7	This experiment
5% KOH mercerized-human hair	122.4	This experiment
80 °C HTH	140.6	This experiment



(**a**) (**b**) (**c**) (**d**)

Figure 4. Contact angle of sea water to the surface of: (a) dry raw Human Hair; (b) 5% NaOH Mercerized Human Hair; (c) 5% KOH Mercerized Human Hair; and (d) 80 °C HTH

3.4. Oil-Water Sorption Analysis

The oil sorption analysis involved of measuring raw and modified human hair's oil sorption capacity in a 20% oil-seawater mixture across three contact times (60, 80, and 100 minutes). The result presented in

Figure 5 including those for raw human hair and various modifications (5% NaOH mercerized hair, 5% KOH mercerized hair, and 80°C HTH). Despite lower oil sorption compared to theoretical values, the modified hair generally showed lower capacity, except for 5% NaOH mercerization at 100 minutes. The raw hair had shown a higher performance of oil sorption for a shorter period of adsorption time compared to others modified hair, while become in efficient for a longer adsorption time. This indicate that the raw hair the structural integrity of the hair has been reduced without any modification done. Thus, it could not sustain longer and performance for a longer adsorption time. At 80 minutes, 5% NaOH mercerized hair performed best, followed by 5% KOH mercerized and 80°C HTH. Notably, the oil sorption capacity of 5% NaOH mercerized hair was higher compared to the control on the 100 minutes, while 80°C HTH shows a similar pattern despite not passing the control oil sorption capacity.

This may indicate that the optimum contact time for some modifications may increase in the oil-sea water mixture (Prem Kumar *et al*., 2022). Nevertheless, this high oil sorption by both 5% NaOH mercerized hair and 80°C HTH were due to their higher hydrophobicity compared to other as proved in the wettability test previously. At 100 minutes oil sorption, the raw hair shows a decreasing in sorption as compared to the 5% NaOH mercerized hair due to the ineffectiveness of raw material without alkaline treated (ease to degrade) and less surface area (Barbora *et al*., 2002). As stated by Vaišis et al. (2022), mercerization had positive effect on absorption capacity by caused swelling and incensement of surface area that led to improved diesel absorption.

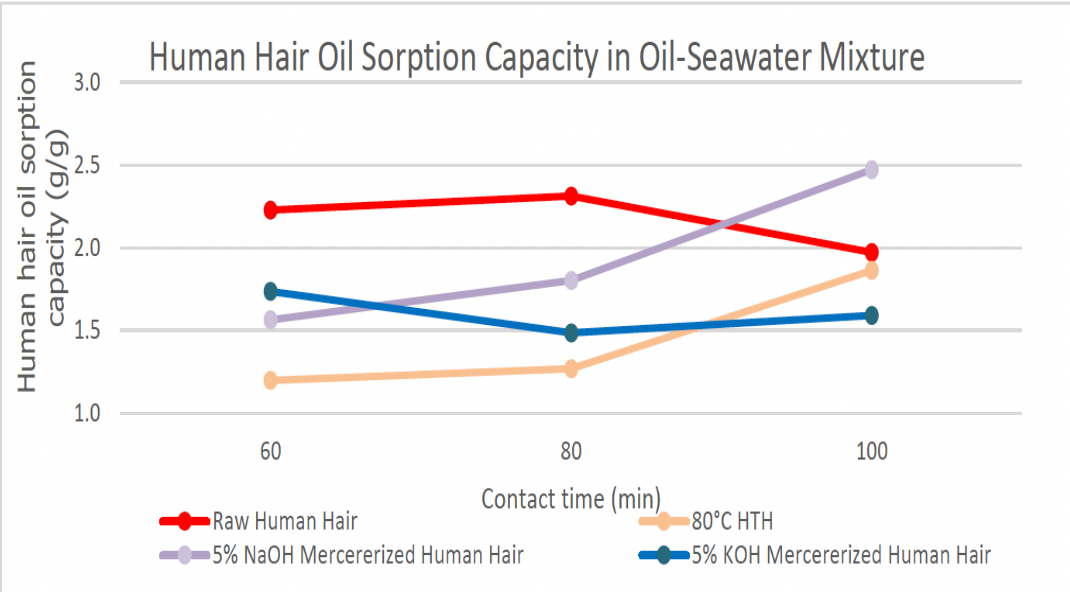


Figure 5. Oil sorption capacity in g/g of human hair sample in oil-seawater mixture

Based on the result achieved from

Figure 5, NaOH mercerization and hot water treatment were chosen for the next variation in HTH temperature and concentration of NaOH for the oil sorption performance at 60 to 100 minutes of adsorption time. These two samples were chosen as both showed an increasing oil sorption as the sorption time increased compared to other samples. This proves the sustainability and reliability of the modified sorbent (HTH and mercerization) in a longer time of adsorption in rough seawater environment. For the mercerization modification evaluation, the concentration was varied from 5% to 35% whereas for the HTH, the temperature was varied from 70 to 100 °C. The result obtained for the concentration varied vs oil sorption is shown as in

Figure 6.

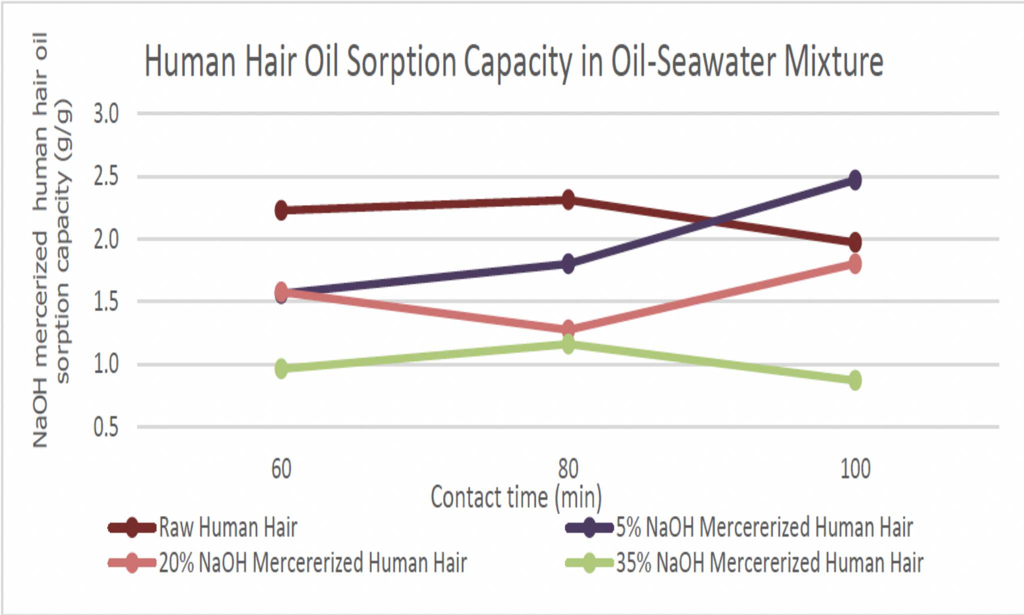


Figure 6. Oil sorption capacity in g/g of 5%, 20% and 35% NaOH mercerized human hair in oil-seawater mixture

Based on

Figure 6, it displayed that increasing NaOH concentration (5%, 20%, 35%) reduced oil sorption, likely due to hair surface damage at higher concentrations. The increased NaOH concentrations seem to contribute to a decrement in oil sorption capacity. This result may be caused by the high concentration values of 20% and 35% NaOH concentrations which have caused more too severe damage to the human hair surface. According to Chavan (2012), cotton alkaline mercerization was usually maxed at 18% to avoid damaging the biosorbent adsorbent characteristics towards liquid. Hence, the best concentrations for 5% NaOH human hair from this experiment were concluded as 5%. **Figure 7** shows the result obtained for the HTH tempe

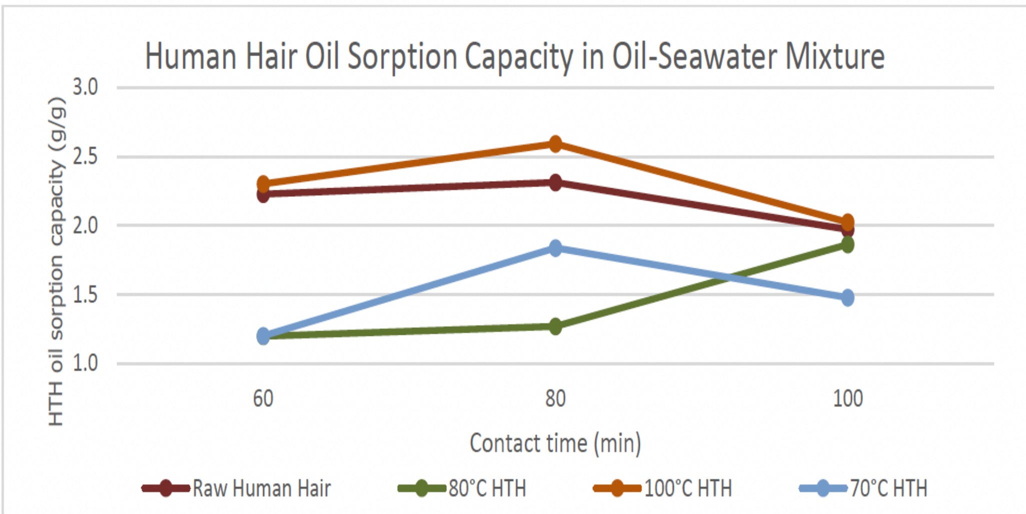


Figure 7. Oil sorption capacity in g/g of 70°C, 80°C, and 100°C HTH in oil-seawater mixture

The highest oil sorption achieved with 2.592 g/g by the 100°C hot water treated sorbent at the adsorption of 80 minutes. It also showed that, the 100°C HTH achieved higher oil sorption capacity as compared to other samples for each of adsorption variation. By comparing to the result obtained from the mercerization process (

Figure 6), it was noted that at 100°C hot water treatment, the oil sorption capacity was higher as compared to NaOH mercerization (2.471 g/g). It shows that, the hot water treatment is much significant to give a good performance in oil sorption capacity compared to the mercerization method. This could be due to the highest contact angle produced (highest hydrophobicity) and surface roughness through the hot water treated hair. Thus, the temperature of 100°C hot water treatment appeared most effective for enhancing oil removal by modified human hair, and it could be varied more for the temperature range and others related parameters to see the further impact. As stated by Vaišis et al. (2022), hot water modification process eliminates the volatile compounds and waxy coatings from the cellulose surface, making it more accessible for the absorption medium, thus increasing sorbent surface area is due to the impact of hot water on sorbent swelling.

4. Conclusion

The modification of the raw human hair with hot water treated and mercerization process were successfully conducted through the improvement of oil sorption factors of surface roughness and hydrophobicity that shown the structural characterization (FESEM/EDX and wettability test) analysis. Based on the oil sorption capacity analysis, it showed that the highest oil sorption, 2.592 g/g adsorption capacity achieved by the human hair-hot water treatment at 100°C with 80 minutes of adsorption time, followed by 5% NaOH mercerization-human hair at 100 minutes of adsorption time with 2.471 g/g adsorption capacity. Through the oil sorption analysis on variation adsorption time, it showed that the optimum adsorption time occurred at 80 minutes with average increment in oil sorption for all the samples tested. These findings suggest that the human hair sorbent show more potential in oil sorption enhancement by treated with hot water. Hot water treatment shows the improvement in the surface roughness and hydrophobic properties (highest contact angle) as compared to the effect of mercerization, that lead to enhancement in oil sorption capacity. The raw human hair showed effectively oil sorption at the short period of adsorption time, and further degrade for a longer time, which result in a lower sustainability in oil-seawater environment. In addition to hot water treatment, it is suggested and could be beneficial in term of sorption capability and efficiency by using combination of hot water and mercerization with NaOH at lower concentration (5-18%) for the human hair modification. Apart from that, a preliminary study on oil sorption in oil-water mixture by the human hair modification need to be conducted before the assessment in oil-seawater mixture as to see the effect of salt towards the sorption efficiency and modified sorbent performance. It is expected that the combine modification at the optimum value could improve surface roughness, hydrophobicity and the structural integrity for the maximum oil sorption for oil-seawater clean-up.

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Credit Author Statement

Conceptualization, Nordin, N.; methodology, Aineh, N.A., Nordin, N.; validation, Nordin, N. and Abdullah, N.; formal analysis, Aineh, N.A.; investigation, Aineh, N.A.; resources, Nordin, N.; writing—original draft preparation, Aineh, N.A.; writing—review and editing, Nordin, N. and Aineh, N.A.; supervision, Nordin, N.; project consultation, Abdullah, N.

Conflicts of Interest

The authors declare no conflict of interest.

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